

**Sertifikaat**

PATENTKANTOOR

REPUBLIC OF SOUTH AFRICA

DEPARTEMENT VAN  
HANDEL EN NYWERHEID



13/10/516372  
Rec'd PCT/PTO

30 NOV 2003  
PATENT OFFICE  
REPUBLIEK VAN SUID-AFRIKA

DEPARTMENT OF TRADE  
AND INDUSTRY

Hiermee word gesertifiseer dat  
This is to certify that

REC'D 01 JUL 2003

WIPO PCT

the documents attached hereto are true copies of the Forms P2, P6,  
provisional specification and drawing of South African Patent Application  
No. 2002/4382 in the names of Highveld Steel and Vanadium  
Corporation Limited

Filed : 31 May 2002  
Entitled : Process

**PRIORITY  
DOCUMENT**  
SUBMITTED OR TRANSMITTED IN  
COMPLIANCE WITH RULE 17.1(a) OR (b)

Geteken te  
Signed at

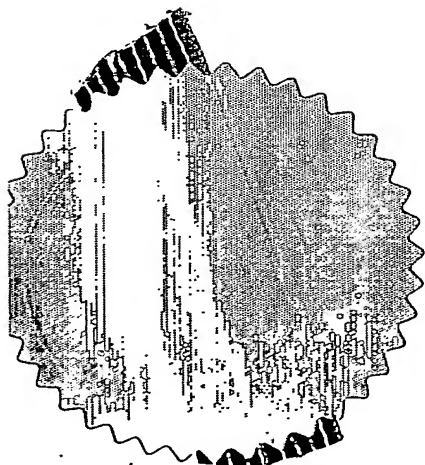
PRETORIA

in die Republiek van Suid-Afrika, hierdie  
in the Republic of South Africa, this

30th

dag van  
day of

May 2003



Registrateur van Patente  
Registrar of Patents

BEST AVAILABLE COPY

REPUBLIC OF SOUTH AFRICA			REGISTER OF PATENTS			PATENTS ACT, 1978		
OFFICIAL APPLICATION			LODGING DATE: PROVISIONAL			ACCEPTANCE DATE		
21	01	2002/4382	22	31 MAY 2002		47		
INTERNATIONAL CLASSIFICATION			LODGING DATE: COMPLETE			GRANTED DATE		
51			23					
FULL NAME(S) OF APPLICANT(S)/PATENTEE(S)								
71	HIGHVELD STEEL AND VANADIUM CORPORATION LIMITED							
APPLICANTS SUBSTITUTED:						DATE REGISTERED		
71								
ASSIGNEE(S)						DATE REGISTERED		
71								
FULL NAME(S) OF INVENTOR(S)								
72	1. DORMEHL, ANDRIES GERHARDUS 2. MONAGHAN, PATRICK ALBERT							
PRIORITY CLAIMED		COUNTRY		NUMBER		DATE		
N.B. Use International abbreviation for country (see Schedule 4)		33	NIL	31	NIL	32	NIL	
TITLE OF INVENTION								
54	PROCESS							
ADDRESS OF APPLICANT(S)/PATENTEE(S)								
OLD PRETORIA MAIN ROAD, WITBANK, SOUTH AFRICA								
ADDRESS FOR SERVICE						S & F REF		
74	SPOOR & FISHER, SANDTON					PA133218/P		
PATENT OF ADDITION NO.				DATE OF ANY CHANGE				
61								
FRESH APPLICATION BASED ON				DATE OF ANY CHANGE				

## APPLICATION FOR A PATENT

AND ACKNOWLEDGEMENT OF RECEIPT  
(Section 30 (1) - Regulation 22)

R 006000

HASR 505

The granting of a patent is hereby requested by the undermentioned applicant on the basis of the present application filed in duplicate

OFFICIAL APPLICATION NO.

21 01 2002/4382

REPUBLIC OF SOUTH AFRICA  
S & F REFERENCE

PA133218/P

FULL NAME(S) OF APPLICANT(S)

71 HIGHVELD STEEL AND VANADIUM CORPORATION LIMITED

ADDRESS(ES) OF APPLICANT(S)

OLD PRETORIA MAIN ROAD, WITBANK, SOUTH AFRICA

TITLE OF INVENTION

54 PROCESS

THE APPLICANT CLAIMS PRIORITY AS SET OUT ON THE ACCOMPANYING FORM P.2. THE EARLIEST PRIORITY CLAIM IS:

COUNTRY: NIL NUMBER: NIL DATE: NIL

THIS APPLICATION IS FOR A PATENT OF ADDITION TO PATENT APPLICATION NO.

21 01

THIS APPLICATION IS A FRESH APPLICATION IN TERMS OF SECTION 37 AND IS BASED ON APPLICATION NO.

21 01

THIS APPLICATION IS ACCOMPANIED BY:

- ☒ 1. A single copy of a provisional specification of 5 pages.
- ☒ 2. Drawings of 1 sheet.
- ☐ 3. Publication particulars and abstract (Form P.8 in duplicate).
- ☐ 4. A copy of Figure of the drawings (if any) for the abstract.
- ☐ 5. Assignment of invention.
- ☐ 6. Certified priority document.
- ☐ 7. Translation of the priority document.
- ☐ 8. Assignment of priority rights.
- ☐ 9. A copy of the Form P.2 and the specification of S.A. Patent Application No.
- ☐ 10. Declaration and power of attorney on Form P.3.
- ☐ 11. Request for ante-dating on Form P.4.
- ☐ 12. Request for classification on Form P.9.
- ☒ 13. Form P.2 in duplicate.
- ☐ 14. Other.

74 ADDRESS FOR SERVICE: SPOOR &amp; FISHER, SANDTON

Dated: 31 May 2002

  
 SPOOR & FISHER  
 PATENT ATTORNEYS FOR THE APPLICANT(S)

REGISTRAR OF PATENTS  
TRADE MARKS AND DESIGNS

RECEIVED  
2002-05-31

REGISTRATEUR VAN PATENTE  
HANDELSMERKE EN OONTWERPE

REGISTRAR OF PATENTS

REPUBLIC OF SOUTH AFRICA  
PATENTS ACT, 1978

## PROVISIONAL SPECIFICATION

(Section 30(1) – Regulation 27)

OFFICIAL APPLICATION NO.

21	01	2002/4382
----	----	-----------

LODGING DATE

22	31 MAY 2002
----	-------------

## FULL NAMES OF APPLICANTS

71	HIGHVELD STEEL AND VANADIUM CORPORATION LIMITED
----	---

## FULL NAMES OF INVENTORS

72	DORMEHL, ANDRIES GERHARDUS MONAGHAN, PATRICK ALBERT
----	--

## TITLE OF INVENTION

54	PROCESS
----	---------

### BACKGROUND TO THE INVENTION

THIS invention relates to a process for the preparation of a vanadyl sulphate solution.

It is known to produce vanadyl sulphate by dissolving vanadium pentoxide in hot dilute sulphuric acid under vigorous agitation and continued heating with the aid of sulphur dioxide as a reducing agent.

The limited solubility of sulphur dioxide in acidic and aqueous solutions results in the emission of sulphur dioxide from the solution and this presents an environmental hazard. Overdosing of the solution with  $\text{SO}_2$  gas results in the unwanted formation of the lower valent vanadium sulphate, namely  $\text{V}_2\text{SO}_4$  and not vanadyl sulphate ( $\text{VO}\text{SO}_4$ ).

Since the dissolution of vanadium pentoxide in sulphuric acid is endothermic heat has to be provided to drive the formation of vanadyl sulphate.

There is thus always a need for a new method for the production of vanadyl sulphate.

### SUMMARY OF THE INVENTION

According to the invention a process for producing a vanadyl sulphate solution includes the steps of:

- (1) providing a starting material comprising vanadium trioxide ( $V_2O_3$ );
- (2) contacting the vanadium trioxide with an appropriate volume and concentration of a sulphuric acid solution to produce a vanadium trioxide suspension; and
- (3) contacting the vanadium trioxide suspension with a strong oxidising agent that is capable of raising the valency or oxidation state of the vanadium, thereby to dissolve the vanadium trioxide in the sulphuric acid to produce the vanadyl sulphate solution ( $VO SO_4$ ).

Various strong oxidising agents including peroxides and permanganate such as hydrogen peroxide, sodium peroxide and potassium permanganate, for example, can be used. Hydrogen peroxide is particularly preferred as it does not introduce any impurities into the final product.

The hydrogen peroxide is typically added slowly to the vanadium trioxide suspension due to the violent nature of the reaction.

### BRIEF DESCRIPTION OF THE DRAWING

The invention will now be described in more detail, by way of example only, with reference to the accompanying drawings in which:

**Figure 1** is a graph indicating the mass relationship between varying quality  $V_2O_3$  expressed as  $V_2O_5$  against constant 4.5g  $V_2O_5$  portions in a 4.0 molar sulphuric acid solution; and

**Figure 2** indicates the reduction potential in mVolt against the mass of the  $V_2O_3$  used.

### DESCRIPTION OF A PREFERRED EMBODIMENT

The crux of the invention is to use a strong oxidising agent to dissolve vanadium trioxide ( $V_2O_3$ ), commonly referred to as Hivox, in a sulphuric acid solution to produce vanadyl sulphate ( $VOSO_4$ ).

In carrying out the process, a strong oxidising agent such as a peroxide or permanganate, for example, is used to dissolve the  $V_2O_3$  in a warm sulphuric acid solution with constant stirring. Although various strong oxidising agents such as hydrogen peroxide, sodium peroxide, potassium permanganate, iodine, potassium iodate, potassium bromate, bromine, ammonium persulfate, persulfates of sodium and potassium, cerium (IV) sulphate, and potassium dichromate, for example, can be used, hydrogen peroxide is preferred as it does not introduce any impurities into the final product.

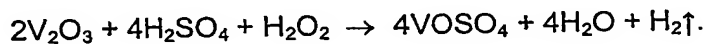
As the quality of industrial grade Hivox ranges typically from 115 to 122 percent equivalent  $V_2O_5$ , figure 1 can be used in order to determine an appropriate quantity of  $V_2O_3$  for use in the process, depending on the quality of the starting material used. The required solution can be obtained by monitoring the reduction potential thereof in mVolt, as shown in figure 2, during the dissolution process. The start of production of vanadyl sulphate is illustrated at the point where the graph dips sharply.

The invention will now be illustrated by way of the following non-limiting example.

**Example**

Hydrogen peroxide was used to dissolve 3.0 grams Hivox ( $V_2O_3$ ) in a warm ( $50^\circ C$ ) 4.0 molar sulphuric acid solution with constant stirring. The hydrogen peroxide was added dropwise as it reacted violently with the solution. During the dissolution process the reduction potential of the solution was continuously monitored and the addition of hydrogen peroxide stopped when the solution reached the end point at 600 mVolts.

The process proceeded according to the following formula:



During the process, the solution first turned green, which is indicative of the presence of  $V^{3+}$  ions, whereafter it turned blue, which is indicative of the presence of  $VO^{2+}$  ions present in the vanadyl sulphate end product.

From the above, it is evident that vanadyl sulphate can readily be made using Hivox ( $V_2O_3$ ), which is generally more cost effective, and less hazardous, than the conventional process using  $V_2O_5$ .

DATED THIS 31<sup>st</sup> DAY OF MAY 2002



SPOOR & FISHER  
APPLICANT'S PATENT ATTORNEYS



200 2 / 4 3 8 2

Figure 1

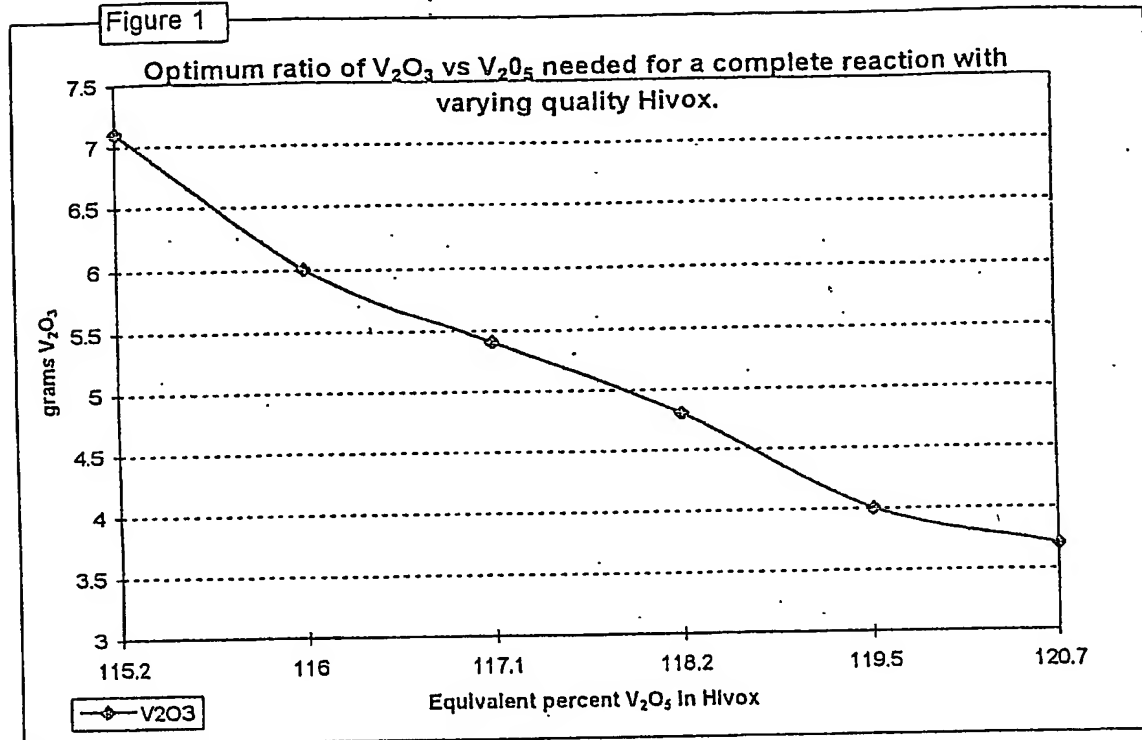
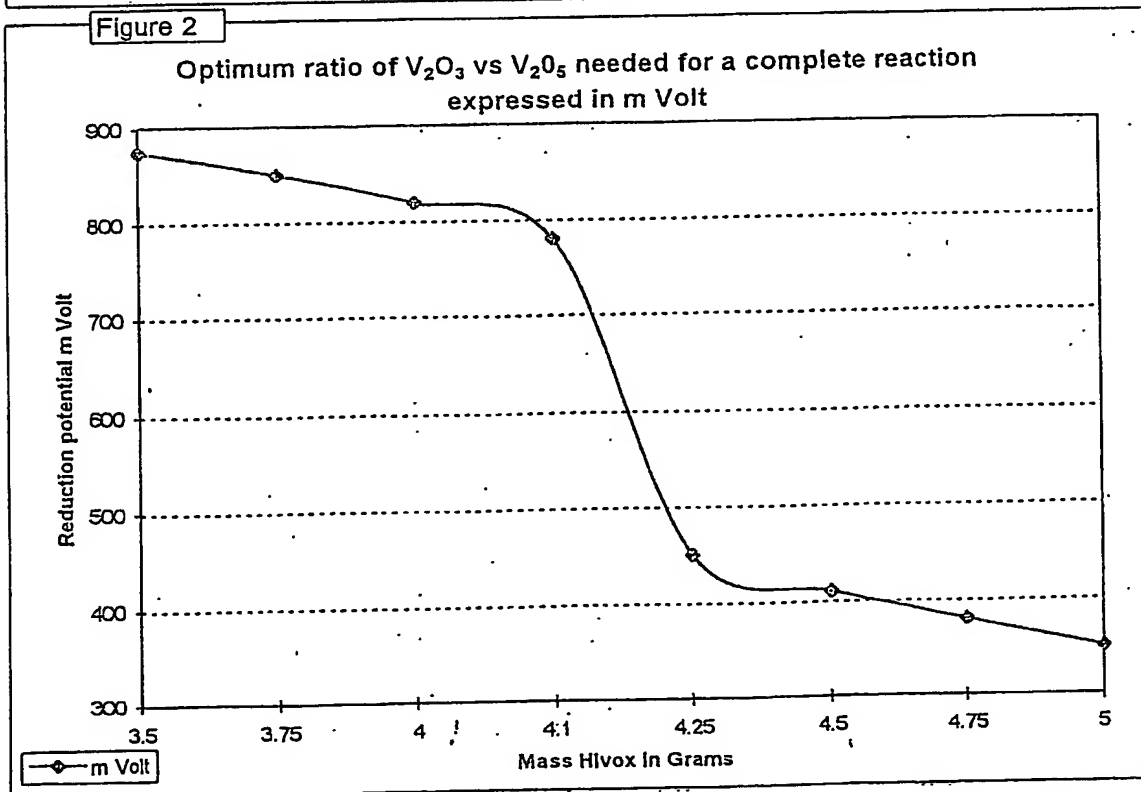


Figure 2



*[Signature]*  
SPOOR & FISHER  
Applicant's Patent Attorneys